

SANDIA NATIONAL LABORATORIES
CHEMICAL & DISPOSAL ROOM PROCESSES DEPARTMENT 6748
WASTE ISOLATION PILOT PLANT PROJECT

TOP-536

CALIBRATION, USE, AND MAINTENANCE OF
THE ATOMSCAN-25 INDUCTIVELY COUPLED PLASMA EMISSION SPECTROMETER

Revision 0

Effective Date: 1/5/96

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1.0 REVISION HISTORY

This document replaces TOP-6119-02 draft 2. The only purpose for this revision is to comply with SNLA-WIPP QA requirements.

2.0 PURPOSE

This procedure provides for the calibration, operation, maintenance of the Atomscan 25 Inductively Coupled Argon Plasma Atomic Emission Spectrometer (ICP-AES) as part of the laboratory geochemistry research activities in support of the Waste Isolation Pilot Plant (WIPP) Project.

3.0 SCOPE

This procedure is applicable only for the Atomscan 25.

This document is not meant to substitute for the manufacturer's instruction manual for the Atomscan 25. The user is responsible for reading and understanding the manual (see references).

4.0 SAFETY

This document does not address ES&H issues. Laboratory ES&H procedures described in the SOP of the lab in which the equipment is used shall be adhered to.

5.0 RESPONSIBILITIES

The Principal Investigator (PI), or designee, whose activities warrant the use of this procedure is responsible for implementing the requirements of this procedure.

The Project Scientist (PS), or designee, is responsible for performing the calibrations and measurements following the requirements of this procedure, documenting calibrations, and assuring that the latest revision of this document is followed.

The Quality Assurance Manager (QA Manager) is responsible for monitoring the work to assure proper implementation of the procedure and for assuring its continued effectiveness.

6.0 CONTROLS

Controls are established by written procedures or instructions prepared in accordance with QAP 5.3, PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program. Procedures are issued in accordance with QAP 6.1,

DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95) of the Sandia National Laboratories WIPP Quality Assurance Program.

7.0 REAGENTS AND STANDARDS

Calibration and standardization will be verified using commercially obtained standards that are traceable to National Institute for Standards and Technology (NIST) or other nationally recognized standards whenever possible. The lot numbers and expiration dates (if any) of the standards used shall be recorded in the laboratory notebook.

The standards will not be used past the expiration date listed on the container by the manufacturer.

Quality Control (QC) samples (performance tests) may be prepared from either commercially obtained standards or may be made by dissolving reagents (see section 7.3).

All standards used (NIST-traceable or not) must have their stated values verified through analysis of a QC sample that has been obtained from an independent source.

7.1 WATER QUALITY

Water of sufficient quality, similar to ASTM Type II reagent water, shall be used for preparing standards and samples, such that calibration blanks and sample blanks (see section 7.4) will produce concentrations at or below instrument detection limits for all analytes of interest. The resistivity of the water shall be greater than 17.5 MΩ.

7.2 ACIDS

All acids used in the preparation of standards and sample processing shall be ultra-high purity grade.

7.3 STANDARDS

Preferred: Purchase certified aqueous stock standards from a supplier and verify by comparison with second standard.

Alternative: Prepare stock standard solutions from reagent grade materials (dried for one hour at 110° C unless otherwise specified). The expiration date of these reagent stock solutions will be one year from the date that they were prepared. This expiration date shall be listed on the container. All stock reagents shall be kept at room temperature to prevent precipitation.

7.4 BLANKS

Two types of blanks are required for the analysis. The calibration blank is used in establishing the analytical calibration curve while the method blank (or preparation

blank) may be used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

7.4.1 The calibration blank shall be prepared by diluting 20 mL of concentrated HNO₃ to 1000 mL with water. The calibration blank is used for instrument calibration (zero concentration standard) and flushing the system between standards and between samples.

7.4.2 The method blank (or preparation blank) must contain all reagents and in the same amounts as used in processing the samples. The method blank shall be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

8.0 QUALITY CONTROL

All measurements must be within the instrument's linear range (for the wavelength of interest) where interference correction factors are valid.

If a sample is analyzed and is found to be outside the instrument's demonstrated linear range, the operator may either 1) Extend the demonstrated linear range by successfully quantifying a standard of higher concentration than the sample in question or 2) Dilute the sample down to within the demonstrated linear range. If neither of these options is viable, see section 8.1.

Linear response is defined as having an r^2 value of 0.995 or greater.

Analyze a minimum of one method blank for each 50 samples prepared.

If results of a QC Sample are not within the control boundaries (± 10 percent), all samples bracketed by this QC sample shall be flagged on the data reports and corrective action documented with the data.

Additional quality control (i.e., duplicates, spikes, additional method or matrix blanks, tighter tolerances, holding times, QC Charts, etc.) is governed by the analytical requirements of the project or specific analyses requested.

8.1 FREQUENCY OF CALIBRATION

For most instruments, the terms *calibration* and *standardization* are interchangeable. Not this one.

Calibration, as used herein (and in the operator's manual), is used instead of standardization in situations where the reference standards used exhibit non-linear response, and the options listed in section 8.0 are not viable.

As per the operator's manual (page 5-31), if a two point linear standardization or a multipoint linear standardization (1 to 5 standards per element) is used, then calibration is not necessary. Since, for most elements, the Atomscan-25 has a linear response range of several order of magnitude, linear standardization is almost always preferable. Calibration will be performed only when it is impractical to dilute the samples to within the linear response range.

When calibration is deemed necessary, it shall be performed daily, immediately prior to analysis.

When calibration is deemed necessary, it will be performed immediately after the failure of a performance test (see section 8.4)

8.2 FREQUENCY OF STANDARDIZATION

Standardization as used herein (and in the operator's manual), refers to situations where the reference standards used exhibit linear response.

Standardization will be performed daily, immediately prior to analysis.

Restandardization will be performed immediately after the failure of a performance test (see section 8.4).

8.3 FREQUENCY OF PERFORMANCE TESTS

A performance test shall be performed immediately after standardization and immediately before sample analysis, as well as immediately after sample analysis. If a batch of samples is being analyzed, a performance test shall also be done once per every five samples.

8.4 PERFORMANCE TEST CRITERIA

Performance tests shall be done by analyzing a QC sample. A difference between the value of the measurement and nominal value of the QC sample of greater than 10% shall constitute a failed performance test.

8.5 CORRECTIVE ACTION

If a performance test is failed, the instrument shall be restandardized or recalibrated. If it still fails, the peristaltic pump should be checked and the hose replaced or reseated if needed. If it still fails, periodic maintenance as described in Appendix 3 shall be implemented, and the performance test shall then be repeated. If the performance test is failed once again, troubleshooting as described in section 19 of the operator's manual shall be consulted and corrective action taken as required. If the instrument still fails its performance test, it shall be tagged and taken out of service until repaired.

Failures of performance tests and the remedial action taken shall be documented on the analysis printout.

Failures of more than one performance test in a given day and remedial actions taken shall be documented in the appropriate scientific notebook.

8.6 GAS REQUIREMENTS

The minimum certified purity of argon to be used is 99.9%.

9.0 CALIBRATION

Calibration will be performed in accordance with section 5.4 of the operator's manual (see appendix 2).

10.0 STANDARDIZATION

Standardization will be performed in accordance with section 4.1.1 of the operator's manual (see appendix 1, page 1-5).

11.0 PROCEDURE: ANALYSIS

Analysis will be performed in accordance with section 4.1 of the operator's manual (see appendix 1, page 1-10).

11.1 TOLERANCES

Tolerances for all measurements made during an analysis shall be specified as follows: 1) a tolerance limit may be stated with a measurement value given in a method, or 2) if a tolerance limit is not stated with a measurement value, then the following system of tolerances shall be in effect:

- a. When one or more significant figures are given to the right of the decimal point, the tolerance limit is ± 0.5 of the least significant digit. The maximum number of significant figures for this method is three.
- b. All class "A" glass pipettes shall be considered sufficiently accurate (1% or better) for use without verification, provided that a visual inspection of the pipette reveals no evidence of breaks or chips to the glass tip or other obvious damage.
- c. Mechanical or electronically operated pipettes shall be verified by the user, before and after single or repetitive deliveries, by weighing at least one aliquot of water. The equivalent volume determined by the weight of the aliquot measure shall be within the manufacturers stated accuracy, typically 0.8% or better, for volumes greater than 500 μL .

11.2 SUMMARY OF METHOD

ICP-ES is a technique for the sequential multi-analyte determination of inorganic analytes in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Aqueous or solubilized solutions of solid samples are nebulized and the aerosol that is produced is transported to a plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled argon plasma (ICP). The spectra are dispersed by a grating spectrometer and intensities of the lines monitored by photomultiplier tubes. Photocurrents from the photomultiplier detector tubes are processed by a computer system.

11.3 INTERFERENCES

A *wavelength scan* (see section 5.1.8.1 of the Operator's Manual) shall be performed prior to analyzing any matrix/analyte combination for the first time to check for interferences.

Standards shall always be matrix-matched.

Room temperature instability effects can affect instrument response. This shall be minimized by "analyzing" deionized water for a minimum of 30 minutes prior to analysis (this will also clean the nebulizer and thus reduce effects caused by salt buildup on the nebulizer tip).

11.4 STARTUP

The Atomscan 25 is normally left in "standby" mode when not in use. If the unit has, for some reason been shutdown, it shall be turned on and left in standby mode for at least 16 hours prior to use.

To prepare the Atomscan 25 for use:

1. Make sure the vacuum pump is running (it is normally left on).
2. Turn on the high-voltage power supply, the cooling unit, the exhaust ventilation (set to at least 200 CFM), the computer and the printer. Make sure the secondary exhaust (located on top of the main unit is not obstructed).
3. To load the program, type: "25" and press "enter".
4. Use the arrow and enter keys to tour through the menu. Select "Plasma Control Panel" under "Setup" and press "enter".
5. Press "F8". Wait for messages to stop flashing (about 30 seconds).
6. Press "escape". Select "Wavelength Calibration" and press "enter".
7. Press "F1". Wait until Wavelength Calibration is completed (about 7 minutes). Press "enter" to accept the calibration.
8. Clamp down the windings to the peristaltic pump (located behind the door in main unit).
9. Select "Plasma Control Panel", and press "enter".
10. Press "F1", then press "F9", then press "enter" to light torch (this will take about 1 minute).

11. Press "escape". Select "Analysis" under "Operation". Press "enter".
12. Select a method, and press "enter".
13. Place sampling tube in deionized water and wait 30 minutes before beginning standardization and analysis.

11.5 SHUTDOWN

1. Place sampling tube in deionized water and wait at least 5 minutes prior to exiting "Analysis" (to flush acids and salt residue out of the system).
2. Select "Plasma Control Panel" under "Setup" and press "enter".
3. Press "F7", then press "enter".
4. Press "escape", then select "Quit to DOS" under "Exit". Press "enter".
5. Turn off High Voltage Power Supply, cooler, exhaust, computer, printer.
6. Unclamp peristaltic pump windings.

12.0 MAINTENANCE

Maintenance will be performed on the instrument as instructed in the operator's manual in section 11(see Appendix 3).

13.0 QA RECORDS

All data, calibrations, standardizations, and performance test printouts, as well as certificates for standards and argon used will be submitted to the SWCF or will be recorded in the laboratory notebook in accordance with Sandia National Laboratories WIPP Quality Assurance Program Procedure 20-2, "PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS" (Revision 1, effective date: 7/31/95).

14.0 REFERENCES

Environmental Protection Agency, 1986. *SW-846, Method 6010: Inductively Coupled Plasma Atomic Emission Spectroscopy*, Office of Solid Waste and Emergency Response, Washington, DC.

Greenberg, A.E., L.S. Clesceri, A.D. Eaton, 1992. *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association, Washington, DC

Thermo Jarrell Ash Corporation, 1991. *Atomscan 25 Spectrometer Operator's Manual*, Thermo Jarrell Ash Corporation, Franklin MA (located in building 823, room 2084)

Wagner, J.J., 1991. *PNL-ALO-211: Determination of Elements by Inductively Coupled Argon Plasma Atomic Emission Spectroscopy*, Pacific Northwest Laboratory, Battelle Memorial Institute, Hanford, WA.

QAP 5.3, PREPARING, REVIEWING, AND APPROVING TECHNICAL OPERATING PROCEDURES (Revision 1, effective date: 7/31/95)

QAP 6.1, DOCUMENT CONTROL SYSTEM (Revision 1, effective date: 7/31/95)

QAP 20.2, PREPARING, REVIEWING, AND APPROVING SCIENTIFIC NOTEBOOKS (Revision 1, effective date 7/31/95)

APPENDIXES

Operator's Manual - Thermo Jarrell Ash Corporation's Atomscan 25

APPENDIX 1:	Analysis	10 pages
APPENDIX 2:	Calibration	13 pages
APPENDIX 3:	Maintenance	13 pages

4.1 ANALYSIS

This module contains the procedures used every day to analyze samples. These procedures include standardization and sample analysis. Daily operation assumes the method to be used has been optimized for the analysis in question and, if necessary, calibrated. These procedures are further discussed in the section entitled Development.

The Analysis module is entered by using the cursor keys to select Operation from the Top Menu, followed by selecting Analysis from the Operation Menu and pressing <Enter>. Immediately upon selecting Analysis, a Method Name will be requested. You may supply this by typing it or by pressing <List> and choosing from the list of available Method names. Once the Method name is supplied, the screen shown in Figure 4-2 will be displayed. This will be the assumed starting point for all procedures described in this Section.

Getting Next Action		Analysis	
WATER			
Command:			
Press a Function Key, or Enter Command and press <Return>		F1= Analyze F2= QC Stndrd F3= Stndrdize F4= Blank F5= Profile F6= F7= F8= ALT Opts F9= Autosmplr F10=HELP	

Figure 4-2: Analysis Main Menu

4.1.1 Standardization

Standardization is the procedure of running known concentration standards to establish a relationship between the raw intensities produced by the instrument and the concentrations which they represent. If the data is to be collected in concentration mode, the instrument must be standardized first.

From the Analysis screen, (Figure 4-2), press the function key labeled <Stdndrize>. The names of all Standardization Standards appear on the screen, as shown in Figure 4-3. These names are obtained from the Method, where a high and a low standard name, or the names of the multipoint standards, should be recorded.

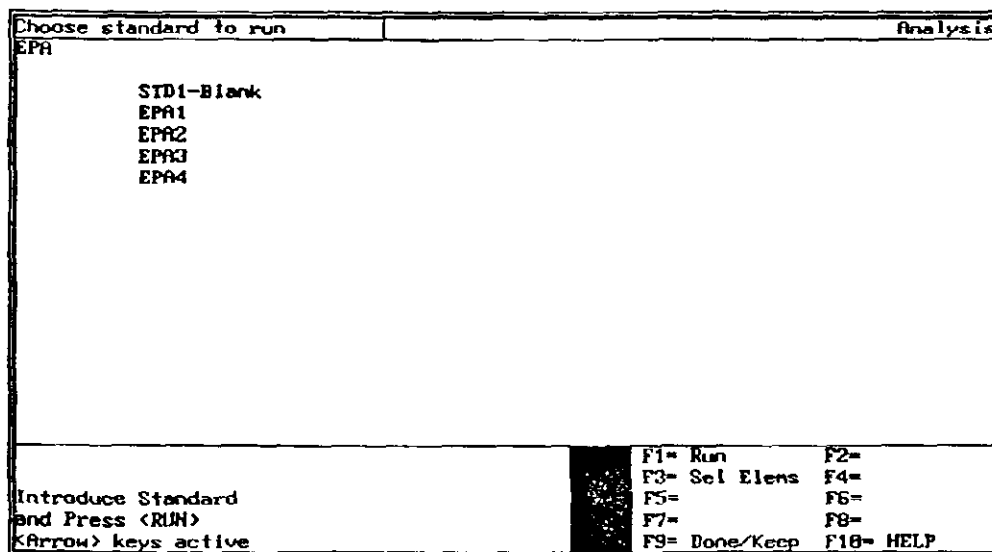


Figure 4-3: Standardization Screen

It is possible to standardize a subset of elements listed in the analytical method. This is done by pressing the function key labeled <Sel Elens> from the Standardization screen (Figure 4-3). A numbered list of elements is presented and you may select by number those elements which are to be standardized. Selected elements are highlighted. When this is completed the list of standards is updated to reflect the new choice.

The standards may be run in any order and need not all be run. After each standard has been run a report like the one shown in Figure 4-4 will be displayed.

Viewing Standardization Data								Analysis
Elem	Ba4554	Ca3933	Ca317s	Cd2265	Co2286	Cu3247	Cu2218	
Avg	43.11	88.86	14.35	28.15	29.77	15.93	.3234	
SDev	.35	.51	.14	.11	.12	.18	.0812	
%RSD	.0086	.6398	.9839	.4877	.3958	.6122	.3638	
#1	42.69	79.53	14.56	28.86	29.66	15.84	.3228	
#2	43.89	79.97	14.38	28.83	29.78	15.89	.3228	
#3	42.65	79.46	14.18	28.87	29.69	15.82	.3238	
#4	42.95	79.93	14.35	28.28	29.85	15.95	.3238	
#5	43.58	88.81	14.31	28.24	29.86	16.18	.3248	
#6	43.25	88.27	14.53	28.28	29.81	15.96	.3248	
#7	43.25	88.25	14.21	28.16	29.77	15.96	.3238	
#8	42.69	79.32	14.27	27.96	29.54	15.78	.3238	
#9	43.36	88.39	14.45	28.25	29.88	15.95	.3268	
#18	43.54	88.69	14.33	28.32	29.91	16.84	.3248	
Press a function key								F1=
								F3=
								F5=
								F7=
								F9= Done/Keep
								F2=
								F4= Print
								F6= Store
								F8=
								F18= HELP

Figure 4-4: Standard Run Report

If the run is accepted, the system returns to the list of standard names. Those which are run and accepted in the current session are flagged with the word "Done".

After the desired standards have been run and accepted, the option is available to select those elements for which the slope and intercept coefficients should be updated (Note that this means you have TWO places where you may select the elements to standardize. Prior to running the standards, you could choose which elements' standards to run. After running them, you can still choose whether or not to update the standardization with the new data).

The chosen element coefficients are then updated using the last set of readings available whether they were obtained now or earlier by pressing <Done/Keep>.

A report, shown in Figure 4-5 is presented, showing the new data.

Viewing Standardization Report						Analysis
Slope = Conc (SIR)/IR						Page 1 of 2
Element	Wavelength	High std	Low std	Slope	Y-intercept	Date Standardized
Cu324s	324.754	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Ba455s	455.483	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Al167s	167.081	STD3	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Be313s	313.042	STD3	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Na589s	589.592	STD3	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Mg279s	279.553	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
K_766s	766.490	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Ca393s	393.366	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
U_389s	389.311	STD5	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Cr267s	267.716	STD4	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Mn257s	257.618	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Fe259s	259.948	STD3	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Co228s	228.616	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Ni231s	231.604	STD3	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Zn213s	213.856	STD2	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
P_177s	177.499	STD4	STD1-Blank	1.00000	.000000	NOT STANDARDIZED
Press a function key						F1= Restdize F2= Print
(Page) keys active						F3= F4= Readback
						F5= SlopeUnits F6= Plot
						F7= F8=
						F9= Done/Keep F10= HELP

Figure 4-5: Standardization Report

From this screen it is possible to go back to the standardization screen if desired, by pressing <Restdize>. The standardization report may be printed by pressing <Print>. The standardization readback may be viewed by pressing <Readback>. The readback report includes known, measured and residual concentrations, along with a correlation factor calculated when multipoint standardization has been set up. See the section entitled "Development" for more information on multipoint standardization.

Signal vs. concentration plots of the standardization data may be viewed by pressing <Plots>. Note that signal represents an intensity ratio. For more information on intensity ratios, see the section entitled "Calculations".

Also, the units of the displayed slopes may be toggled by pressing <SlopeUnits>. The default standardization slope units may be set in the SDT table by toggling the option "Default Standardization Slope Units:" See the section entitled "Configuration" for more information on the SDT table. The option "Conc(SIR)/IR" is the default. Slope will be defined as being the ratio between Concentration (or Standardized Intensity Ratio if Calibration is being used), and Intensity Ratio (Intensity divided by the internal standard which may be real, time or none). In this case, the Y-Intercept will also be displayed. This is the value of the intercept on the Concentration axis, and will usually be negative. The absolute value of this number represents the Background Equivalent Concentration (BEC). The option "IR/Conc(SIR)" will cause the slope to be defined as being the ratio between Intensity Ratio, and (Concentration or Standardized Intensity Ratio). In this case, The Background Equivalent Concentration (BEC) will be displayed which is the absolute value of X-Axis (Concentration) intercept. This option is for Display purposes only, and will have no effect on calculations or analytical data results.

If the data is acceptable, pressing the function key labeled <Done/Keep> will save the new coefficients in place of the previous values.

The decision as to how often to standardize depends on how much drift is taking place and how much accuracy is desired. This can be established by periodic running of QC Check samples. For analysis of aqueous solutions of low solids content in a laboratory where stable temperature and humidity conditions exist, a standardization once a day may be adequate. Good practice would dictate that you standardize anytime analytical conditions are altered. Extreme cases would require standardization once every few samples. Normally, once every 2 hours is enough.

4.1.2 Analysis of Samples

Once the instrument has been standardized, it is ready to analyze samples. From the Analysis screen (Figure 4-2), the following procedures may be carried out (in addition to Standardization described above). Additional operating instructions are available at every stage by pressing <Help>.

Three types of samples may be identified; unknowns, QC Check samples and blanks. While analysis of each is carried out in the same way, each type may contain its own name and comments and is subjected to separate check Tables.

- a. Press <Analyze> to initiate analysis of an unknown sample. You may enter information about the sample before initiating the reading. The procedure is detailed below. Results appear on the screen and are printed to selected devices in the specified format. If Limit Checking is enabled, the Limit Check Table specified in the Method is used.
- b. Press <QC Stndrd>, to initiate analysis of a QC Check sample. This is available in Concentration Mode only. QC Check samples are checked against the QC Check Table specified in the Method. You may also use the result of the analysis of the QC Check sample to adjust the slope OR intercept coefficients. This is further discussed under the section entitled "Normalization".
- c. Press <Blank>, to initiate analysis of a Blank. Raw data collected for a Blank sample will be retained in the computer memory. If Blank Subtraction is activated, the LATEST VALUES are subtracted from the results of all subsequent unknowns. The values for the LAST BLANK are always used for Blank Subtraction.

Whenever <Analyze>, <QC Stndrd>, or <Blank> is pressed, the screen shown in Figure 4-6 is displayed.

Setting Sample Information		Analysis	
WATER Unknown			
Output Mode = Concentration		Blank Subtraction = No	
Background Correction = Yes		Limit Checking = No	
Limit Check Table = LCTAB		Override Significant Figures? = No	
Override Print Limits? = No			
Sample Name : Comment : Operator ID : Corr. Factor : 1 Cust. ID : Cust. Samp. ID : Lab. ID :			
Introduce Unknown and Press <RUN>			
Enter Sample Name		F1= Run	F2= Output
(up to 20 characters)		F3=	F4=
<Arrow> keys active		F5=	F6=
		F7=	F8=
		F9=	F10=Help

Figure 4-6: Editing Sample Information

At this point you may type in sample information using the arrow keys to select the information field, or you may use the function keys.

Press <Output> to change the output options specified in the Method. The new conditions will affect all subsequent runs of the APPROPRIATE SAMPLE TYPE (i.e. samples, QC Checks or Blanks, depending on which function key was last pressed) until you change the conditions OR exit the Analysis Mode. When you return to Analysis, the Default conditions will once again be in effect. The unknown output screen is shown in figure 4-7.

Modifying Output Parameters		Analysis	
DATE: Unknown			
Output Mode:	Concentration		
Override Print Limits?	No		
Override Significant Figures?	No		
Apply Background Correction?	Yes		
Apply Blank Subtraction?	No		
Limits Table: LCTAB	Check?	No	
Correction Factor:	1		
Report to: Screen	1. Aves	2. Stats	3. Reps
Printer	4. Errs	5. Units	6. Int.Stds
<div style="display: flex; justify-content: space-between;"> <div> <SPACE> to view options <CR> to select option <Arrow> keys active </div> <div> F1= F3= F5= F7= F9= Done/Keep </div> <div> F2= F4= F6= F8= F10=HELP </div> </div>			

Figure 4-7: Editing Analysis Output Information

NOTE: This option is NOT AVAILABLE when operating in the PROTECTED Mode. See the section entitled "Development" for more information on the protected mode.

Press <Run> to initiate running the sample. Note that during the measurement the label on this key changes to <Abort Run>. Press this key during the run to terminate the data collection process.

4.1.3 Modifying Analysis Setup Conditions

A function key labeled <Setup> is available under the ALT function key definitions which allows the modification of the conditions for future analysis.

Press <ALT Options> to redefine the regular function keys. A new set of labels will appear. Once you become accustomed to where these ALT options are used, these labels may also be obtained by holding the <ALT> key while pressing the appropriate function key. The available ALT function keys will change depending on the data available.

Press <Setup> to change the Setup parameters. The new conditions will affect all subsequent runs until you change the conditions OR exit the Analysis Mode. When you return to Analysis, the Default conditions will once again be in effect. The setup screen is shown in figure 4-8. The setup questions are described in detail in the section entitled "Development".

Changing Setup Options		Analysis	
WATER			
Number of Repeats:	4		
Flush Time:	30.8		
Auto-increment samplenames?	No		
Auto-store Analysis Data?	No		
Auto-store Stdzn Data?	No		
Store Individual Repeats?	No		
Auto-Print Analysis Data?	No		
Auto-Print Stdzn Report :	None		
Condensed Print Format?	No		
Collect scans?	No		
Auto-Print Scans?	No		
Minimum Number of Repeats is 1		F1=	F2=
Maximum Number of Repeats is 10		F3=	F4=
<Arrow> keys active		F5=	F6=
		F7=	F8=
		F9= Done/Keep	F10=Help

Figure 4-8: Editing Analysis Setup Information

NOTE: This option is NOT AVAILABLE when operating in the PROTECTED Mode. See the section entitled "Development" for more information on the protected mode.

Changes made to conditions when using the ALT options DO NOT affect the Default conditions specified in the Method. Changes made here are temporary and are in force only during the current analysis session.

4.1.4 Manipulating Analysis Data

Because the last set of raw data is always retained in memory, the capability exists to modify the analytical conditions and then recalculate the results based on these new conditions. This is begun by pressing the function key labeled <ALT Options>. You will notice that there are now many more options than existed before the first sample was run. Many of the NEW options refer to the data that was just generated, NOT to data to be collected in the future. The following options are available.

- a. Press <Esc> to return the function keys to their NORMAL definitions.
- b. Press <Output> to change Output options on a TEMPORARY basis for the CURRENT DATA ONLY. You may change any of the parameters and then press <Done/Keep> to recalculate the results based on the new conditions. Results for the NEXT analysis will be reported based on the original output parameters. These are established after selecting the sample type, just prior to pressing <Run>.
- c. Press <Print> to PRINT the results of the LAST analysis using the CURRENT Output options.
- d. Press <Store> to STORE the results of the LAST analysis using the CURRENT Output options.

4.1.5 QC Normalization

There is an additional alternate option which is available ONLY WHEN A QC CHECK SAMPLE HAS BEEN RUN. This allows use of the QC result to modify the standardization slope OR intercept. ONLY ONE OF THESE MAY BE MODIFIED. WHENEVER ONE IS SELECTED, THE OTHER IS IMMEDIATELY RETURNED TO ITS DEFAULT VALUE. To use this feature, press <Normalize>. The options <Slope>, <Y-Intrcpt>, and <FromTable> appear.

Now press <Slope> to normalize the SLOPE. A factor is computed which adjusts the standardization slope (i.e. rotates the standardization line about the intercept) to force the QC Check sample to read the correct value. Details of the calculation may be found in the section entitled "Calculations". All elements will have their slopes normalized.

Press <Y-Intrcpt> to normalize the INTERCEPT. A factor is computed which adjusts the standardization intercept (i.e. moves the standardization line up or down, keeping the slope constant) to force the QC Check sample to read the correct value. Details of the calculation may be found in the section entitled "Calculations". All elements will have their intercepts normalized.

Press <FromTable> to normalize on an element by element basis depending on the information entered in the QC table.

4.1.6 Selecting a Subset of Elements for Analysis

The alternate function key labeled <Sel Elems> will allow the selection of a subset of elements listed in the current method for use in future analysis. A list of available elements will appear. Toggle the selection of elements by number.

Note, in order for the concentration calculations to be completed correctly, any elements required for special corrections such as interfering element corrections must be selected as well.

This feature may also be used with data in memory to display or print only a subset of the elements collected.

4.1.7 Autosampler Operation

This option is available only if the instrument is configured with an autosampler.

From the Analysis screen (Figure 4-2) press <Autosampl>. You will then be prompted to enter an autosampler table name. You may enter a name or select from a list of available names. These functions are fully documented with Help screens.

You may select a terminating action to be performed at the end of the autosampler run. These options include none, alarm, and shutdown. The alarm consists of an audible signal which will be issued until a key is pressed. The shutdown option is available for instruments with automatic plasma control. A full system shutdown will occur at the completion of the autosampler run.

You may then begin the run at the beginning of the table by pressing <Run>, or you may select another starting point within the table using the cursor keys. This allows you to halt a run and begin it again in the same place.

The system will check to see if the autosampler is available. If the Autosampler is NOT available, the system will open a warning window and ask if you wish to proceed manually. If you press <Enter> (i.e. "yes"), you will enter the Software Supervised Operation mode. In this mode, the operator will be prompted for the samples, standards, and QC checks to be run based on the previously created autosampler Table. This mode is more fully described in the section entitled "Advanced Operations".

5.4 CALIBRATION

To access the Calibration options select Development from the top main menu. The three calibration options are shown in figure 5-15. If a two point linear standardization, or a multipoint linear standardization (1 to 5 standards per element) is to be used, then calibration is not necessary.

Calibration is required only if you wish to use non-linear calibration curves, more than five standards per element, or you wish to calculate multiple order IEC correction factors.

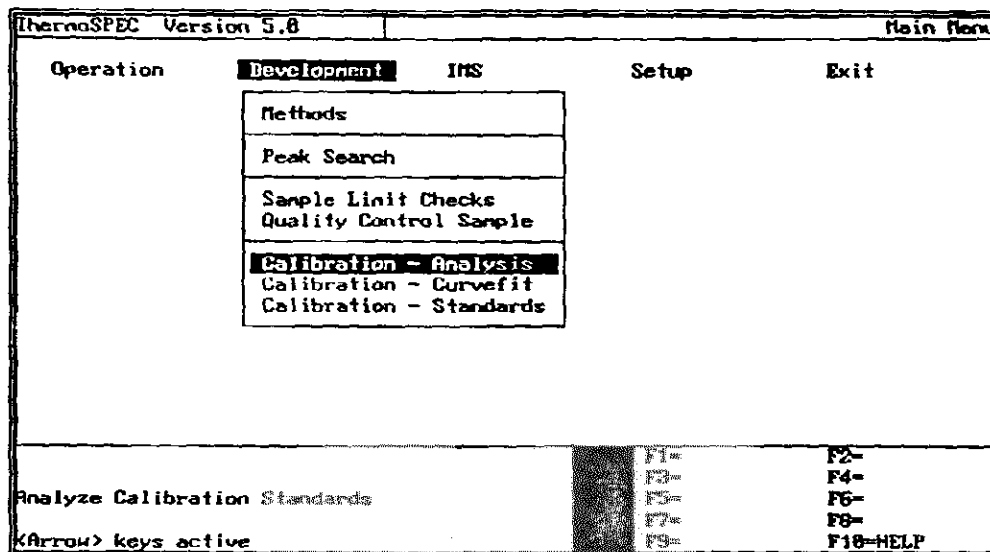


Figure 5-15: Calibration Operations

Three operations are available with Calibration. The first, Analysis, allows you to analyze the calibration standards and store the data. After analysis has been performed, the second calibration function, Curvefit, is used to fit the standards data to a calibration curve. Before analysis can be performed, the third function, standards, must be used to enter the calibration standard information into a Table.

5.4.1 Analysis of Calibration Standards

Analysis of calibration standards is accessed by selecting "Calibration - Analysis" from the Development Menu. In response, you are requested to provide a method name, which refers to the analytical method to be calibrated. Type in the method name and press <Enter>, and in response the name of the Calibration Standards Table and the Calibration Data File, as entered in the method, are displayed on the screen. You may calibrate the method using a standards table other than that entered in the method, and you may store the calibration data to a file other than that entered in the method; to do so, type in the names of the standards table and data file that you wish to use. Alternately, of course, you may use the table and file entered in the method, in which case no change is required. When you are satisfied with the standards table and data file selections, press <Done>, and the calibration standards from the appropriate Calibration Standards Table are displayed on the screen shown in Figure 5-16.

Calibration		Calibration - Analysis	
Method : WATER	Standards : WATER_S	Calib Dat : WATER_D	
Page 1 of 1			
1. Blank 2. Standard 2 3. Standard 3 4. Standard 4 5. Standard 5			
<Arrow> keys active		F1= RunStdnd	F2= Setup
		F3= Norm Std	F4=
		F5=	F6=
		F7=	F8=
		F9= Done	F10=HELP

Figure 5-16: Calibration Standards to be Analyzed

To obtain analytical data, press the vertical arrow keys to place the cursor over the standard that you wish to run, and press <RunStdndrd> to initiate analysis. Data will be automatically stored to the appropriate data files. Repeat this procedure until all the calibration standards have been analyzed, then press <Done> to return to the Calibration Menu. Note that data will be appended to old data files. If you wish to keep your data separate, you must specify a new data file.

Other functions available under Analysis of Calibration Standard are the following:

1. Press <Setup> to change the setup parameters, flush time, integration time, printout or display parameters.
2. Press <Profile> to perform an instrument profile.
3. Press <Norm Std> to run a Normalization Standard. THE STANDARD CURRENTLY HIGHLIGHTED WILL BE RUN AS A NORMALIZATION STANDARD. The purpose of the normalization standard is to eliminate the effects of any drift by tracking a single standard which is run at the beginning and then periodically during the calibration run. In effect, the system is normalizing all the data to "time zero"; the time when the selected standard was first run. The first data collected for the selected standard becomes the Original Value. Whenever <Norm Std> is subsequently pressed and the standard is run, the new data is used to compute a factor.

Eq. 5-1

$$f = \frac{\text{Original Value}}{\text{Latest Value}}$$

All subsequent calibration standards data is modified by this factor before being stored.

NOTES: <Norm Std> need NOT be pressed to establish the original value. So long as the selected standard has been run when the key is first pressed, the ORIGINAL VALUE WILL BE EXTRACTED FROM THE CALIBRATION DATA FILE.

If Standard Normalization is to be used, the selected standard should always be run FIRST to establish the "zero time" to which all other standards will be.

- Press <Sel Stds> to select a subset of standards to be run. A numbered list of standards is presented. Make a selection by entering the numbers of the desired standards. Note that a choice can be made simply by placing the cursor over the desired standard and pressing <Run>, then repeating this procedure for the desired standards. There is no requirement that all standard names appearing on the screen be analyzed.

5.4.2 Calibration Curvefit

The calibration curvefit function is accessed by selecting "Calibration - Curvefit" from the Development Menu. In response you are requested to provide the name of the method to be calibrated. Type in the method name and press <Enter>, and in response, the names of the Calibration Standards Table and the Calibration Data File as entered in the method are displayed on the screen. If you have used the standards table and data file from the method to collect data, simply press <Done>; however, if you used another standards table or data file to collect the data during analysis, then type in the appropriate names before pressing <Done>. In response, the elements in the method to be calibrated are displayed on the screen along with information concerning the curvefit last applied to each element; the screen shown in Figure 5-17 shows an example for a one element method which has been curvefit.

Calibration Fit Status			Calibration - Curvefit	
400STNLS	Mode: Conc. Ratio	Standards: 400STNLS	Date: 400STNLS	
No. of Elements: 11		Page 1 of 2		
		No. of Elements Selected: 11		
El-Name	Wavelength	Date-of-Fit	Type-of-Fit	Correlation
C	193.893+	28 Jun 87 08:58	Curvilinear	.999757
Mn	293.386+	28 Jun 87 09:03	Linear	.997919
P	178.287+	28 Jun 87 09:07	Linear	.997382
S	188.781+	28 Jun 87 09:11	Linear	.995784
Si	251.612+	28 Jun 87 09:21	Curvilinear	.999726
Cu	327.396+	30 Jun 87 01:57	Linear	.971475
Ni	243.789+	28 Jun 87 09:38	Linear	.999682
Cr	298.919+	30 Jun 87 01:47	Curvilinear	.998123
Mo	282.838+	30 Jun 87 02:03	Curvilinear	.999942
V	318.238+	2 Apr 88 03:23	Linear	1.000000

Use the arrow keys to select next Element to Fit <Arrow> keys active		<Page> keys active	
F1= Fit Elen	F2= Print	F3= LinFitall	F4=
F5= Sel Elen	F6= InfoAvail	F7=	F8=
F9= DoneFit	F10=HELP		

Figure 5-17: Elements to be Curvefit

Use the vertical arrow keys to select the element that you wish to fit, then press <Info Avail> to obtain a summary of the data available to the curvefit routine for the highlighted element, as shown in Figure 5-18.

Information Available			Calibration - Curvefit	
400STNLS	Mode: Conc. Ratio	Standards: 400STNLS	Data: 400STNLS	
			Page 1 of 1	
Type of Fit Possible	# of Standards	# of Data Points	Element Name	Wavelength
Full Fit	7	7	C	193.093*
Full Fit	7	7	Mn	293.306*
Full Fit	7	7	P	178.287*
Full Fit	7	7	S	180.781*
Full Fit	7	7	Si	251.612*
Full Fit	7	7	Cu	327.396*
Full Fit	7	7	Ni	243.789*
Full Fit	7	7	Cr	298.919*
Full Fit	7	7	Mo	282.638*
Full Fit	7	7	V	310.230*
Full Fit	7	7	Co	345.351*
			F1=	F2= Print
			F3=	F4=
			F5=	F6=
			F7=	F8=
			F9=	F10=HELP

Figure 5-18: Calibration Information Available to the Curvefit Routine

Next press <Fit Elem> to begin the curvefit function for the highlighted element. After the element has been fit, repeat the curvefit procedure until all elements have been fit. The <LinFitAll> function is of special interest in ICP emission spectrometry; it automatically applies a weighted linear regression straight line fit to all the elements in the method. After all the elements have been curvefit, press <Done Fit> to store the curvefit to the method.

Other functions available under Calibration Curvefit are the following:

1. Press <SelElems> to Select Elements on which curve fittings experiments are to be performed. Initially, all elements are shown on the screen and are available for fit trials. By pressing this key, you can limit the list of elements to a chosen subset of all the elements in the Calibration Files. This is useful if you wish to apply <LinFitAll> to several of the elements but you know that it will not work for others. By using this option, you can preserve the current calibration curve for the latter group by locking them out of the current session.

2. Press <Print> to obtain a printout of curvefit data for ALL the elements and lines. A table will be presented offering a variety of options. You may choose to print any or all of them. The options are Table Report, Fit Summary, Readback Report, Linear Plot, and Log Plot.

5.4.2.1 Experimenting with <Fit_Elem>

To experiment with individual elements, use the arrow keys to highlight the desired element name, then press <Fit ELEM>. The screen show in Figure 5-19 will be displayed. This screen shows the current coefficient values for the selected element. If this element has not been calibrated before, the values of A0 and A2 will be 0 while the values of A1 and n will be 1. Warning messages will appear in the lower portion of the screen below the Standard Error of the Fit as appropriate. These will warn of such things as an inflection point or positive A2 (the latter indicating an upward curve). They will not prevent use of the curve if you still wish to do so.

Fitting Calibration Data			Calibration - Curvefit	
400SINLS	Mode: Conc. Ratio	Standards: 400SINLS	Data: 400SINLS	
Element: Cu		Wavelength: 327.396*		
Fit	: Linear	Date of Fit: 30 Jun 87 01:57		
Constant	Value	Error		
A0 (Offset)	: .8955683	(.8319681)		
A1 (Gain)	: 679.8879	(95.88789)		
A2 (Curvature):	0	(0)		
n (Exponent):	1.00	(.88)		
Correlation	: .971475			
Standard Error:	.8389341			
		F1= Calculate	F2= Print	
		F3=	F4= LinPlot	
		F5=	F6= LogPlot	
		F7=	F8= ReadBack	
		F9= Done/Keep	F10=HELP	

Figure 5-19: Curve Coefficients for Selected Element

The following options are now available:

- a. Press <Calculate> to calculate a new calibration curve or to continue experiments with the current element.

- b. Press <Print> to obtain a printout of calibration information concerning THIS element name. A list of options will appear. You may elect to print any or all of them. The options are Fit Summary, Readback Report, Linear Plot, Log Plot, Fit Parameters and Standards Data.
- c. Press <LinPlot> to obtain a Linear plot of standards data according to the current calibration coefficients.
- d. Press <LogPlot> to obtain a Log-Log plot of the standards data according to the current calibration coefficients.
- e. Press <ReadBack> to view the calculated values of the standards according to the current calibration coefficients. This report also shows the given standards concentrations and the percentage difference between given and calculated values.
- f. Press <Done/Keep> to ACCEPT the coefficients and return to selecting elements for further fitting. As soon as you press this key, ALL CONDITIONS USED TO OBTAIN THE COEFFICIENTS WILL BE LOST AT THIS TIME. BE SURE TO PRINT ANY INFORMATION YOU WISH TO RETAIN. When you next press <FitElem>, the coefficients will be restored but standard defaults will be offered for all options. If you wish to experiment further with THE CURRENT ELEMENT, press <Calculate>. The curve you have just calculated together with its conditions will be restored.

5.4.2.2 Calculating a Fit

When you select <Calculate> from Fitting Calibration Data the screen shown in Figure 5-20 is displayed.

Editing Calculation Params		Calibration - Curvefit	
400STNLS	Mode: Conc. Ratio	Standards: 400STNLS	Data: 400STNLS
Element: Cu	Wavelength: 327.396*	#Stds: 7	#Pts: 7 Size: 7
Fit :	Full Fit		
Weight :	None		
IECs?	Yes		
Exponent High :	1.18		
Low :	.68		
Constant	Use-in-Calc		
A0 (Offset) :	Yes		
A1 (Gain) :	Yes		
A2 (Curvature):	Yes		
n (Exponent) :	Yes		
<SPACE> to view options <CR> to select option <Arrow> keys active		F1- EditData F3- EditIECs F5- F7- F9- Done/Calc	F2- F4- F6- F8- F10=HELP

Figure 5-20: Curve Calculation Parameters

Upon first entry, this screen will show the most complex fit which is possible, given the number of standards available. If you re-enter by pressing <Calculate> from Fitting Calibration Data BEFORE you have pressed <Done/Keep>, the latest calculation parameters are shown. You may choose a lesser fit if you wish.

Weighting may be performed if desired using either inverse concentration squared or inverse variance. A "zero factor" allows for the possibility that the normalization parameter may be zero. If this occurs (e.g. for the Blank when using an ICAP), the weight for this point is the zero factor times the smallest non-zero value of the parameter.

The following options are now available:

1. Use the arrow keys to move the cursor around the screen and edit the parameters. With the exception of Exponent High and Low, all are rolling menus with options limited to what is possible, given the number of standards.
2. Press <EditData> to view and edit and the standards data. If you select this option, the screen shown in Figure 5-21 is displayed.

Editing Standards Data			Calibration - Curvefit		
400STNLS	Mode: Conc. Ratio	Standards: 400STNLS	Data: 400STNLS		
Element: Cu			Page 1 of 1		
Wavelength: 327.396*			Size: 7		
#Stds: 7 #Pts: 7					
Standard	Conc. Ratio	Signal	1/V	1/C ²	Emphasis
SS72	.0000000	.8588881	-----	277e6	1
SS71	.0000000	1.173158	-----	277e6	1
SS70	.0000000	.7715286	-----	277e6	1
SS69	.0000000	.7426722	-----	277e6	1
89D	.0006887	.4992488	-----	2771521.	1
92A	.0010662	.8716281	-----	879713.1	1
93D	.0011253	.8169568	-----	789688.9	1
Maximum emphasis is 10			F1= AddStd	F2= Calc C/R	
Use 0 to remove the point			F3= RemStd	F4=	
<Arrow> keys active			F5=	F6=	
<Page> keys active			F7=	F8=	
			F9= Done/Keep	F10=HELP	

Figure 5-21: Standards Data for Current Element

Here you may edit the Emphasis for a standard. This is the number of times the standard will be used in the calculation. A HIGHER emphasis INCREASES the importance of the standard. It does NOT however, change the degree of the most complex curve which can be calculated.

You may also add or remove "User Standards". These are concentration and intensity ratios which you enter manually to evaluate various regions of the curve. Such standards are identified by the names - UserStd00 through -UserStd99 and these names cannot be edited. Note that REAL STANDARDS CANNOT BE REMOVED. They can be temporarily deleted from the calculation however, by setting their EMPHASIS to 0. To add a "User Standard", press <AddStd>. To remove a "User Standard", press <RemStd>.

To see the concentration which would be calculated for any standard, using the current fit coefficients, given the signal value, press <CalConc>.

When you are satisfied with the contents of this screen, press <Done/Keep>. You will return to Editing Calculation Parameters.

Editing IECs		Calibration - Curvefit	
400STNLS	Mode: Conc. Ratio	Standards: 400STNLS	Data: 400STNLS
Element: Cu		Wavelength: 327.396*	
Page 1 of 1			
Interferent :			
k1	:	No IECs selected for this element *****	
k2	:		
Calculate ? :			
Standards :			
		F1= SelIECs	F2=
		F3=	F4=
		F5=	F6=
		F7=	F8=
		F9= Done/Keep	F10=HELP

Figure 5-22(a): IEC Screen with No IEC's selected

Editing IECs		Calibration - Curvefit	
400STNLS	Mode: Conc. Ratio	Standards: 400STNLS	Data: 400STNLS
Element: Cu		Wavelength: 327.396*	
Page 1 of 1			
Interferent :			
k1	:	Mn	P
k2	:	.0000000	.0000000
Calculate ? :			
	:	K1 Only	K1 Only
Standards :			
	:	89D	89D
	:	92A	92A
	:	93D	93D
	:	SS69	SS69
	:	SS70	SS70
	:	SS71	SS71
	:	SS72	SS72
There is no minimum value for k1		F1= SelIECs	F2= SelStds
There is no maximum value for k1		F3=	F4= ShowConc
<Arrow> keys active		F5=	F6=
<Page> keys active		F7=	F8=
		F9= Done/Keep	F10=HELP

Figure 5-22(b): IEC Screen with IEC's selected

- Press <EditIECs> to edit IEC data. One of the screens shown in Figure 5-22 will be displayed, depending on whether any IEC's have already been selected. You may select (or alter the current selection of) IEC's by pressing <SelIECs>.

You may now enter values for k1 and/or k2 or elect to have ThermoSPEC compute these. If you elect to have ThermoSPEC compute the IEC's, you may choose to compute either k1 or k1 and k2.

BE SURE YOU HAVE SUFFICIENT STANDARDS FOR THE NUMBER OF DEGREES OF FREEDOM INVOLVED!

Remember, the minimum number of standards required for each type of curve is as follows:

LINEAR 2 Standards

CURVILINEAR 3 Standards

FULL FIT 4 Standards

In addition, if BOTH k1 and k2 are to be calculated, EACH INTERFERING ELEMENT REQUIRES TWO MORE STANDARDS; one for each degree of freedom (i.e. k1 and k2).

If only k1 is to be calculated, one additional standard is required for each interferent.

DO NOT, UNDER ANY CIRCUMSTANCES, TRY TO DEFEAT THE SYSTEM BY CALCULATING THE IEC COEFFICIENTS SEQUENTIALLY WITH INSUFFICIENT STANDARDS FOR DEGREE OF THE CURVE PLUS THE IEC'S

The standards should contain a variety of concentrations for analyte and interferent and also a variety of ratios of analyte to interferent. The calculation WILL NOT WORK if all standards contain the same ratio of analyte to interferent.

You may also select the standards which will be used for IEC calculation. Press <SelStds> to perform this function.

If there are interfering elements listed, press <ShowConc> to obtain a listing of the standards and concentrations for the analyte element and the HIGHLIGHTED interferent. Use the arrow keys to move the highlight cursor to the desired interferent.

Press <Done/Keep> to accept the current IEC's and return to Editing Calculation Parameters or press <Esc> to return WITHOUT accepting the updated IEC's.

4. Press <Done/Calc> to calculate new coefficients using the set of conditions listed, together with the current IEC's and any "User Standards" and emphasis values entered and accepted. Press <Esc> to return to the previous coefficients without any recalculation. You will be returned to the Fitting Calibration Data screen following the calculation.

NOTE: At this point, the conditions under which the new coefficients were calculated are still retained. If you now press <Calculate> again, the data you just entered will still be present. If however, you press <Done/Keep> on the Fitting Calibration Data Screen, the coefficients will be accepted but the conditions will be lost.

5.4.3 Calibration Standards Table

The calibration standards table allows you to enter the elements and their concentrations contained in calibration standards. This table is required only if you plan to calibrate the spectrometer; if two point standardization is sufficient for your application, then this table is not required.

To access the calibration standards table, select "Calibration - Standards" from the Development Menu. As described in the previous section, you will be requested to supply a name for the standard table.

The name may be either a new name, which will create a new table, or it may be the name of a previously entered table, which will cause the table to be recalled from memory for editing, and display a screen similar to the one shown in Figure 5-23.

Selecting Standards		Calibration - Standards	
Standards table: WATER		Page 1 of 1	
1: Blank	(c)		
2: Standard 2	(c)		
3: Standard 3	(c)		
4: Standard 4	(c)		
5: Standard 5	(c)		
Press a function key		F1= Edit Std	F2= PrintTable
<Arrow> keys active		F3= Add Std	F4=
		F5= Del Std	F6=
		F7=	F8= CopyStd
		F9= Done/Keep	F10=HELP

Figure 5-23: Calibration Standards Table

Entries into the calibration standards table are made by pressing <AddStd>, and you will be requested to name the standard. After naming the standard and selecting the calibration mode, press <Enter>, then press <Elements> and the periodic table will be displayed. Select the elements to be included in the standards and press <Done>. Then type in the concentrations for the elements selected and press <Done/Keep>. This completes the entry for the first standard; repeat the same procedures for all the calibrations standards you will need, then press <Done/Keep> to store the calibrations standards table to memory.

SECTION 11: MAINTENANCE

This section describes the basic maintenance of the ICAP both in terms of day to day operations and periodic servicing of some components. This maintenance is very important to the performance and long term reliability of the system. Just as with an automobile, lack of regular upkeep will result in inopportune down time and expensive service calls later on. Please read the sections on nebulizer and torch upkeep carefully. Failure to follow the instructions on cleaning and assembling these can and will result in irreparable damage and fairly costly replacement.

Basic maintenance must start with some very basic common sense. This ICAP spectrometer is a very complex and precise instrument that is very often in the company of some very corrosive materials. Do not leave corrosive samples open near electronic components. Always keep marble chips (available very inexpensively at any garden supply shop) in the waste bucket to minimize fumes near the power supply. Never ever keep open samples on top of the instrument or inside the sample introduction compartment. Please keep the door closed to this compartment when not servicing the sample introduction components.

11.1 SAMPLE INTRODUCTION SYSTEM

The sample introduction system on the Thermo Jarrell-Ash ICAP spectrometers has gone through several changes before arriving at the present system which represents the best in both performance and ease of use. The maintenance is very similar for the different designs and the schematics for each design are supplied at the end of this section.

111.1.1 Connecting the peristaltic pump tubing

The following procedure refers to Figure 11-1

1. Press the end of the Teflon uptake tube (B) into one end of the peristaltic pump tube winding (C).
2. Press one end of a narrow piece of Teflon nebulizer tubing (E) into the other end of the peristaltic pump tube winding (C).

3. Insert the pump winding into the holding slots on the peristaltic pump cartridge (D) so that the uptake tubing (B) is closest to the front where the pump is marked with an upward pointing arrow. Place the uptake tubing through the grommet on the door frame so that it may easily reach the sample (A).
4. Press the other end of the connecting Teflon tube (E) into the adapter which then connects to the capillary inlet of the nebulizer (F).

Note: Be careful with the capillary on the cross-flow nebulizer. Do not force any sample tubing onto the capillary as it may move the capillary and ruin the nebulizer.

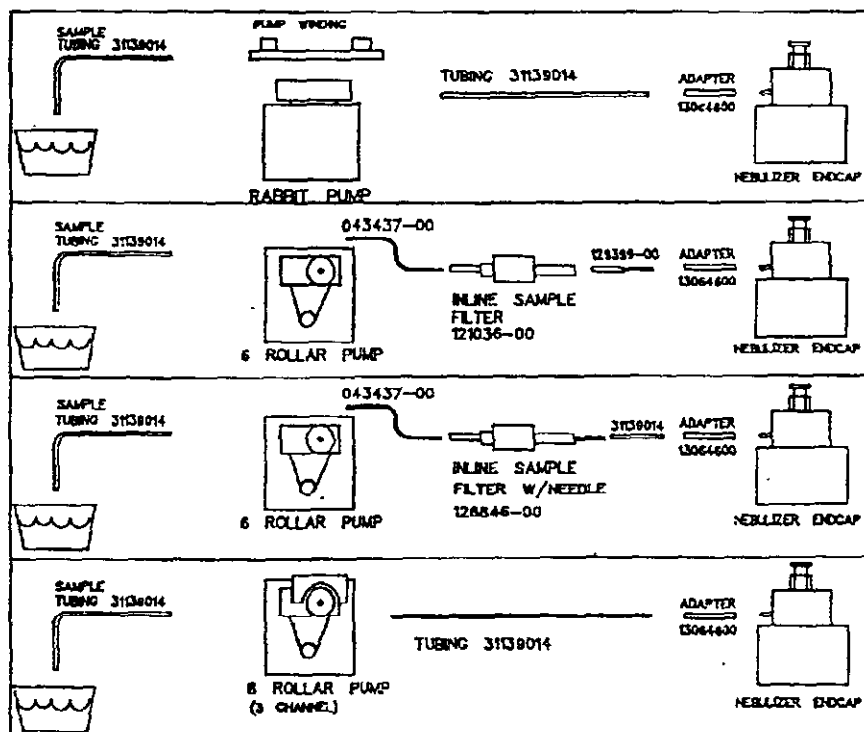


Figure 11-1: Sample Introduction Tubing Diagram

The following figures represent all of the various sample introduction systems available for your instrument. Refer to the figure which represents your sample introduction system for the procedures described in the rest of this section.

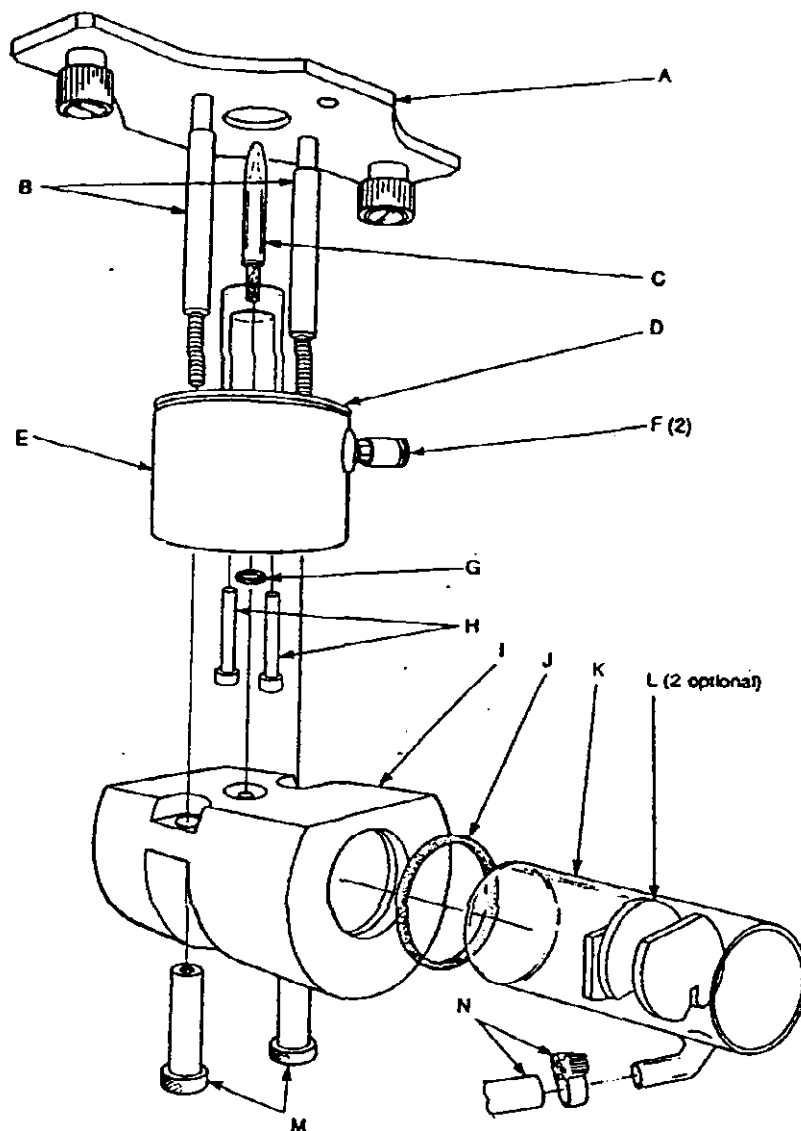


Figure 11-2: Sample Introduction System - Teflon Glass

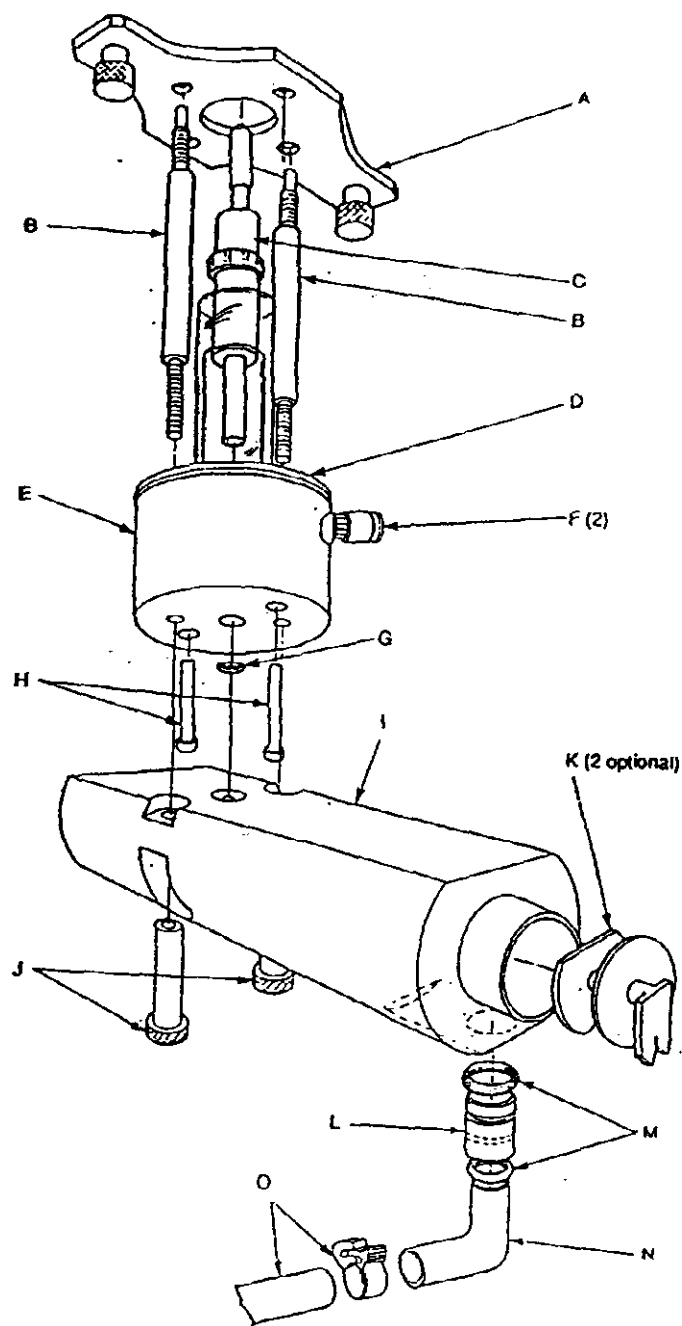


Figure 11-3: Sample Introduction System - HF resistant

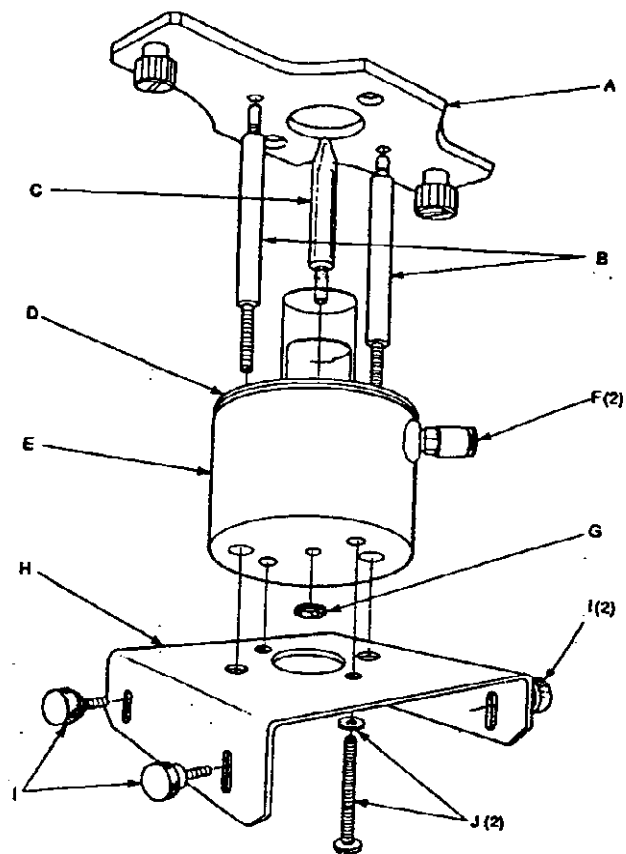


Figure 11-4: Sample Introduction System - Glass (Top)

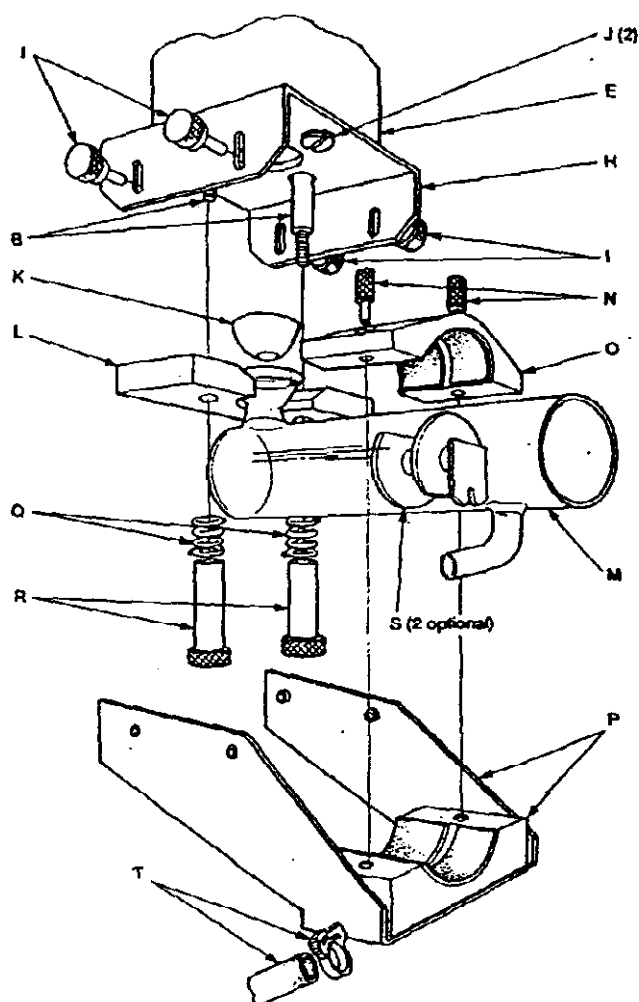


Figure 11-5: Sample Introduction System - Glass (Bottom)

11.1.2 Removing and Cleaning the Nebulizer:

1. Disconnect the peristaltic pump tubing from the pump.
2. Disconnect the argon line to the nebulizer (A) from the inlet on the base of the cabinet.
3. Remove the nebulizer (A) from the mixing chamber (C). The nebulizer press-fit onto the spray chamber, so pull the nebulizer out while twisting to disconnect. Be careful not to bend or damage the capillary while twisting.

At this point, the nebulizer can be placed in an ultrasonic cleaner and left to soak for ten minutes. If you do not have an ultrasonic cleaner, then follow the instructions below:

1. Hold the nebulizer so that the cup or open end is upward.
2. Fill the nebulizer cup with distilled water.
3. Insert a syringe into the end of the capillary tubing and draw back pulling the distilled water through the nebulizer.
4. Repeat step three or four times.
5. Reconnect the nebulizer to the mixing chamber and connect the argon supply

Alternatively: Disconnect the capillary tubing from the nebulizer and clean the nebulizer with a cleaning wire only when absolutely necessary as it is easy to damage the capillary. These wires are available from TJA. (P/N 90-974B001)

Alternatively:

1. Leave the argon supply connected.
2. Place a small amount of distilled water in the nebulizer cup.
3. Turn on the Nebulizer Sample Gas to about 20 psi.
4. Place your hand firmly over the nebulizer cup causing the sampling gas to force the liquid back through the capillary tubing.
5. Repeat three or four times.
6. Reconnect the nebulizer to the mixing chamber.

When operating properly, the nebulizer will freely aspirate distilled water when operated at 32 psi or 0.65 L/m. Disconnect the sample tubing from the peristaltic pump winding so that only the short piece connecting to the nebulizer capillary remains. When set up as above the free aspiration rate should be about 1.5 to 2.0 mL/m. If the rate is substantially less than this, check for obstructions in the capillary and in the argon orifice. This is done by removing the fitting on the back of the nebulizer and blowing out the cavity with a pressurized stream of air or argon. Obstructions can be seen with a magnifying lens or dissecting microscope.

Refer to Figure 11-6 for information pertaining to connecting the nebulizer gas line.

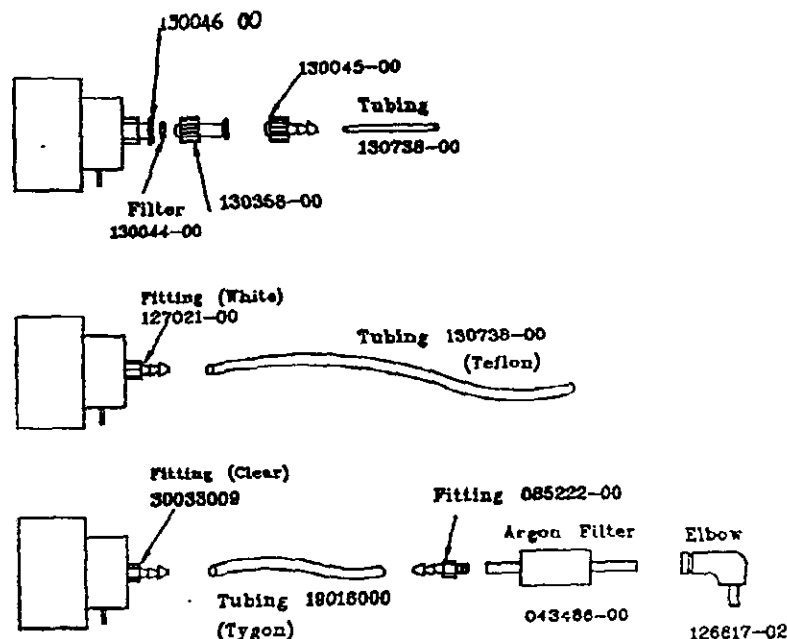


Figure 11-6: Nebulizer Argon Fittings

11.1.3 Removing the Teflon/Glass Mixing Chamber and Drain:

1. Disconnect the nebulizer (A) from the mixing chamber (C).
2. Remove the drain line from the glass elbow (D) on the mixing chamber. Be sure to lower the liquid level in the line first.
3. Remove the two thumb screws (E) on the bottom of the mixing chamber.
4. Slide the mixing chamber down, and remove.
5. The baffle(s) (B) may be pulled out of the mixing chamber with an allen wrench or a paper clip bent and hooked over the baffle.

11.1.4 Cleaning the Mixing Chamber:

The mixing chamber may need to be cleaned if it develops oily film deposits that cause the sample to bead and form large droplets on the chamber walls. The sample should drain smoothly from the sides of the chamber.

To clean the mixing chamber, wash with soapy water. The mixing chamber does not need to be dried before re-installation in the spectrometer. The easiest way to remove any oily film in the glass spray chamber is to aspirate a 1% HF acid solution for several minutes. Keep in mind that this will cause a high Si blank for a while afterwards. Also be very careful in handling Hydrofluoric acid; read the instructions on the bottle carefully. The polypropylene spray chamber can only should be cleaned with soapy water and a brush only. Any film (which will contain some Si) remaining on the inside of the chamber from manufacturing should be removed if the analysis of Si in solutions containing HF is desired. This can only be done by aspirating relatively concentrated solutions (10 - 20%) of HF for several hours.

11.1.5 Removing the Plasma Torch:

1. Remove the mixing chamber (C).
2. Remove the plasma chamber door by loosening the four thumb screws holding it to the housing.
3. Disconnect the gas tubing from the push fittings on the ceramic base of the torch (I).
4. Loosen the two thumb screws (N) on the torch mounting plate (O) which hold the torch to the torch box. Lower and remove the torch.
5. Remove the center sample tube (M) by removing the "O" ring (H), and dropping the center tube out onto a soft surface.
6. Remove the torch mounting plate (O) by loosening the two allen screws (F) on the bottom of the torch ceramic base (I), and removing the screws with their washers (G). Gently lift the mounting plate with the mixing chamber supports (L) off of the torch (K).

11.1.6 Cleaning the Plasma Torch:

The plasma torch must be cleaned whenever salt deposits begin to clog the central sample tube orifice, or whenever metallic deposits cause elevated and non-reliable blank readings. The torch disassembles into two parts, one comprised of the outer and intermediate tubes mounted to the ceramic base, and one comprised of the sample tube. The torch can be cleaned without disassembling, or it may be disassembled and each piece cleaned separately.

To remove salt deposits, wash the torch in an ultrasonic bath for a few minutes using soapy water. Try to avoid getting the ceramic base very wet if possible by suspending the torch upside down in the cleaning solution. Getting the base wet is not a problem as long as the solution is not acidic, but it will necessitate a long drying time.

To remove metallic deposits, dip the tip of the torch into hot acid, either HCL, HNO₃, or a mixture of the two acids.

Note: Extended exposure to acids may damage the epoxy seals in the torch.

After cleaning, rinse the torch copiously with distilled water and dry thoroughly at 95 °C in a drying oven.

11.1.7 Installing and Aligning the Plasma Torch:

1. Insert the center sample tube (M) into the torch so that the bottom slides into the hole in the ceramic base (I). Using a finger to hold the center tube in place from the top (make sure the tube is not hot), push the "O" ring (H) onto the bottom of the center tube until it is against the ceramic base. Failure to set the sample tube down all the way may cause damage to the tip.
2. Lower the torch mounting plate (O) with the spray chamber support screws (L) onto the ceramic base of the torch, and attach with the two allen bolts (F) and washers (G).
3. Insert the torch by sliding it upward through the work coil. Make sure that the argon gas inlets (J) are on the right (as in not left) side.
4. Secure the torch in place with the two thumb screws (N) on the torch mounting plate (O) to the bottom of the torch box.
5. Reconnect the argon gas lines by pushing them firmly into the connectors (J). The connector farthest to the back on the torch, attaches to the connector farthest to the back on the base of the cabinet. The connector farthest to the front on the torch attaches to the middle connector on the base of the cabinet.

11.1.8 Installing the Mixing Chamber and Drain:

1. Make sure that all "O" rings are in good condition, and replace if necessary.

2. Insert the baffle(s) (B) into the mixing chamber (C) so that the v shaped groove on the front is past the drain hole.
3. Slide the mixing chamber onto the supports (L), and position it so that it is snug against the ceramic torch base (I).
4. Secure the mixing chamber with the two thumb screws (E).
5. Replace the drain line on the connecting elbow (D).
6. Replace the nebulizer (A) with pump tubing and argon supply.

11.2 FILTERS

The filters on the back of the power unit and monochromator should be cleaned every few weeks depending on the use and position of the instrument and the cleanliness of the room.

Use the following procedure for a thorough cleaning:

1. Locate the filters on the back of the controller compartment and the back of the source.
2. Pry the filter frames off the panel.

WARNING: THE FANS ARE SPINNING INSIDE THE HOUSING. BE CAREFUL TO AVOID GETTING YOUR FINGERS CAUGHT. TO TURN OFF THE FANS, YOU MUST SWITCH OFF THE MAIN POWER SWITCH.

3. Slide out the square filters located behind the frames.
4. Clean the filter with mild soap in warm water.
5. Rinse and dry the filter thoroughly before replacing it.
6. Set the filter into its mounting rails and press the frames firmly back into place.
7. If you have turned off the main power switch, turn it back on

11.3 MONOCHROMATOR VACUUM SYSTEM

The oil level in the vacuum pump should be checked once a month through the viewing glass on the end of the pump. Refer to the pumps operating manual for instructions on adding oil and changing the filter on the top of the pump, and other necessary maintenance procedures.

The monochromator vacuum pump should be left on at all times. This means that an argon supply should be available to the instrument at all times as well. If it is necessary to turn off the power unit, the vacuum pump should be plugged into another source, or should be disconnected from the spectrometer altogether.

If the argon and vacuum pump are turned off for any extended period the monochromator will require an extended equilibration time during which the analytical peaks may drift.

11.4 REMOVING AND INSTALLING THE MONOCHROMATOR PURGED OPTICAL PATH EXTENSION

1. Extinguish the Plasma.
2. Remove the plasma chamber door by loosening the four thumb screws holding it to the housing.
3. Using the ThermoSPEC control panel, raise the observation height to 40 mm.
4. Loosen and remove the thumb screw which secures the POP tube, located outside and immediately to the right of the torch box.
5. Slide up the POP tube to the left until the threaded mounting stud slides under the stop, and is clear of the mount.
6. Rotate the POP until the stud is pointing downward.
7. Slide the POP the rest of the way out. It may be necessary to push the ignitor out of the way. Just make sure that it is returned to its proper position.
8. Reinstall the POP by reversing the above procedure. Raise the observation height to 40 mm, and position the POP so that the stud rests against the stop. The stop should be positioned so that the end of the POP tube is not over the outer quartz tube of the torch. Lowering the POP tube onto the torch may cause damage.

11.5 CHANGING FUSES

Occasionally you may need to replace the fuses on the power unit distribution panel (see Figure 2-2), or on the optical system side panel (see figure 2-7). Should your instrument blow a fuse, follow the instructions below. Otherwise, you do not need to touch the fuses.

1. **TURN OFF THE MAIN POWER. NEVER TRY TO CHANGE A FUSE WITH THE MAIN POWER ON.**
2. Turn the plastic fuse cap counter clockwise (power unit), or press the bottom of the fuse cap (optical system side panel), and pull it out. The fuse should come with it.
3. Pull the blown fuse out of the cap.
4. Insert a new fuse of the proper current rating into the cap.
5. Push the fuse and cap back into the socket and lock it in place.
6. If the replacement fuse blows, do not replace it with a **third** fuse. Instead, call your TJA Service Engineer.

11.6 COOLANT WATER FILTER

The coolant water filter should be changed approximately every 6 months. The filter may require replacement more or less frequently depending on the quality of the water being passed into it. The life of the filter may be extended when using a closed recirculation system, by using distilled water in the reservoir.

Refer to Figure 2-4(c) for the location of the water filter cartridge. With the water recirculator turned off, use the appropriate size wrenches to remove the old cartridge, and install the new one.